



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION VIII

999 18th STREET - SUITE 500  
DENVER, COLORADO 80202-2466

SDMS Document ID



374980  
403140

Ref: 8ES-MEB

November 6, 1995

**MEMORANDUM**

SUBJECT: Data validation for Rico Argentine Mine Site, Case #24008, SDG # MHDA89

FROM: Russ Leclerc *RL 11/4/95*  
Chemist  
Program Support Group, Technical Support Team

TO: Greg Oberly  
8HWM-SM

The Environmental Services Assistance Team (ESAT) has completed its review of data from the analysis of three water and nine soil samples for Contract Laboratory Program (CLP), Routine Analytical Services (RAS) total metals and cyanide analyses and one water sample for CLP RAS dissolved metals analyses for **Rico Argentine Mine Site, Case 24008, Sample Delivery Group (SDG) #MHDA89**. I have evaluated ESAT's data validation package and agree with ESAT's review. Data in the enclosed package are acceptable with the qualifiers added to the data reports. Please refer to the attached ICF Kaiser data validation report including the narrative summary and comments for a full explanation of the data review findings.

If you have any questions, or if I can be of further assistance, please contact me at 312-6971.

Attachments



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REGION VIII  
RAS INORGANIC - SUMMARY OF CLP DATA QUALITY ASSURANCE REVIEW

CASE	SITE NAME	SITE ID\OPERABLE UNIT
24008	Rico Argentine Mine	8ZZ/00
RPN NAME	ESAT TID - 08-9510-703	
Greg Oberly	ESAT WUD - 26	

LABORATORY	CONTRACT NO.	SDG	LABORATORY TPO/REGION
Southwest Laboratory of Oklahoma	68-D3-0040	MHDA89	Ray Flores/VI

DATA REVIEWER Kristy K. Grove REVIEW COMPLETION DATE 11/07/95

SAMPLE ID	SAMPLE LOCATION	MATRIX	DATE COLLECTED
MHDA89	RA-WSE-02	Soil	09/13/95
MHDA90	RA-WSW-03	Water	09/13/95
MHDA91	RA-WSE-03	Soil	09/13/95
MHDA95	RA-WSE-01	Soil	09/13/95
MHDA97	RA-SO-3	Soil	09/13/95
MHDA98	RA-SO-4	Soil	09/13/95
MHDA99	RA-GW-01	Water	09/14/95
MHDB00	RA-SO-05	Soil	09/14/95
MHDD34	RA-SO-06	Soil	09/14/95
MHDD35	RA-WSO-08	Soil	09/14/95
MHDD36	RA-GW-01	Water	09/14/95
MHDD37	RA-SW-24	Water	09/14/95
MHDD38	RA-SO-07	Soil	09/14/95

*reviewed  
11/6/95  
KLG*

DATA QUALITY STATEMENT\*

- ( ) Data are ACCEPTABLE according to the Functional Guidelines with no qualifiers (flags) by the reviewer
- (X) Data are acceptable with QUALIFICATIONS noted in review
- ( ) Data are UNACCEPTABLE according to the Functional Guidelines

Telephone/Communication Logs Enclosed? Yes \_\_\_\_ No X

TPO Attention Required? Yes X No \_\_\_\_

If yes, list the items that require attention:

Water sample MHDA90 was analyzed outside the technical holding time for cyanide.

\* Please see Data Qualifier Definitions, attached to the end of this report.

## INORGANIC DATA QUALITY ASSURANCE REVIEW

### REVIEW NARRATIVE SUMMARY

This data package was reviewed according to the EPA document "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," February 1994.

Case 24008, SDG MHDA89 consisted of three water and nine soil samples for CLP RAS total metals and cyanide analyses and one water sample for CLP RAS dissolved metals analyses.

Water sample MHDA96 from Case 24008, SDG MHDD70, was a designated rinsate blank and was used to evaluate results for samples collected on 09/13/95. There was no rinsate blank for samples collected on 09/14/95. Results for the 09/13/95 rinsate blank are attached to the end of the report.

The following table lists the data qualifiers added to sample analyses.

SAMPLE ID	ELEMENTS - QUALIFIERS	PROBLEM	REVIEW SECTION
MHDA90	cyanide - UJ	Holding Times	Holding Times
MHDD38	potassium - J	Negative Blank Results	Form B
MHDD35	thallium - J		
MHDA90, MHDD37	aluminum - UJ	Continuing Calibration Blank Results	
MHDA90, MHDA99, MHDD37	thallium - UJ		
MHDA90	aluminum - UJ	Rinsate Blank Results	
MHDA89, MHDA95, MHDA97, MHDA98	antimony - UJ		
MHDA89, MHDA91, MHDA97	beryllium - UJ		
MHDA91, MHDA95, MHDA97, MHDA98	sodium - UJ		
MHDA90	zinc - UJ		
MHDA89, MHDA91, MHDA95, MHDA97, MHDA98, MHDB00, MHDD34, MHDD35, MHDD38	copper, lead, manganese, zinc - J	Matrix Spike Sample Analysis	Form 5A
MHDA89, MHDA91, MHDA95, MHDA97, MHDA98, MHDB00, MHDD34, MHDD35, MHDD38	aluminum, calcium, iron, lead, zinc	Duplicate Sample Analysis	Form 6

## INORGANIC DATA QUALITY ASSURANCE REVIEW

SOW OLM03.0

### RAS INORGANIC DELIVERABLES COMPLETENESS CHECKLIST

P Inorganic Cover Page  
P Inorganic Analysis Data Sheets (Form I)  
P Initial Calibration and Calibration Verification Results (Form II)  
P Continuing Calibration Verification Results (Form II)  
P CRDL Standard for ICP & AA (Form II, Part 2)  
P Blank Analysis Results (Form III)  
P ICP Interference Check Sample Results (Form IV)  
P Spiked Sample Results (Form V)  
P Post-digest Spiked Sample Analysis (Form V, Part 2)  
P Duplicate Sample Results (Form VI)  
P Instrument Detection Limits (Form X - Quarterly)  
P Laboratory Control Sample results (Form VII)  
NA Standard Addition Results (Form VIII)  
P ICP Serial Dilution Results (Form IX)  
P ICP Interelement Correction Factors (Form XII - Quarterly , or Form XI - Annually)  
P ICP Linear Ranges (Form XII - Quarterly)  
P Raw Data  
    P Samples      P Calibration Standards      P Blanks      P Spikes  
    P Duplicates      P ICP QC (ICS and Serial Dilution)      P LCS  
    NA Furnace AA      P Mercury Analysis      P Cyanide Analysis  
P Percent Solids Calculations - Solids Only  
P Sample Prep/Digestion Logs (Form XIII)  
P Analysis Run Log (Form XIV)  
P Traffic Report(s)  
P Chain of Custody  
P Sample Description  
P Case Narrative  
P Method References

KEY: P = Provided in original data package, as required by contract  
R = Provided as Resubmission  
NP = Not provided in original data package or as resubmission  
NR = Not required under contract  
NA = Not applicable to this data package

Comments: None.

## INORGANIC DATA QUALITY ASSURANCE REVIEW

### HOLDING TIMES

All CLP-SOW holding times were met.

Yes ☐ No ☒

All technical holding times were met.

Yes ☐ No ☒

Comments: Water sample MHDA90 was analyzed for cyanide outside the technical holding time of 14 days from date of sample collection. The following table summarizes holding time violations and qualifiers added to the data.

SAMPLE NUMBER	DAYS OUTSIDE LIMITS	ELEMENT	QUALIFIER
MHDA90	1	cyanide	UJ

### INSTRUMENT CALIBRATION: STANDARDS AND BLANKS

Initial instrument calibrations were performed according to contract requirements.

Yes ☒ No ☐

Comments: None.

The instruments were calibrated daily and each time an analysis run was performed.

Yes ☒ No ☐

Comments: None.

The instruments were calibrated using one blank and the appropriate number of standards.

Yes ☒ No ☐

Comments: None.

### FORM 1 - SAMPLE ANALYSIS RESULTS

Sample analyses were entered correctly on the Form I's.

Yes ☒ No ☐

Comments: None.

**INORGANIC DATA QUALITY ASSURANCE REVIEW**

**FORM 2A - INITIAL AND CONTINUING CALIBRATION VERIFICATION**

The initial and continuing calibration verification standards (ICV and CCV, respectively) met contract requirements.

Yes X No   

Comments: None.

The calibration verification results were within 90-110% recovery for metals, 80-120% for mercury, and 85-115% for cyanide.

Yes X No   

Comments: None.

The continuing calibration standards were run at 10% frequency.

Yes X No   

Comments: None.

**FORM 2B - CRDL STANDARD FOR ICP AND AA**

ICP Analysis: Standards (CRI) at 2X the CRDL or the IDL whichever were greater, were analyzed at the beginning and the end of each sample run, or at a minimum of twice per eight hour shift, whichever was more frequent.

Yes X No   

Comments: None.

GFAA Analysis: Standards (CRA) at the CRDL or the IDL whichever were greater, were analyzed at the beginning of each sample run.

Yes    No    N/A X

Comments: None.

The CRI and/or the CRA were analyzed after the ICV.

Yes X No    N/A   

Comments: None.

# INORGANIC DATA QUALITY ASSURANCE REVIEW

## FORM 3 - BLANKS

The initial and continuing calibration blanks (ICB and CCB, respectively) met contract requirements.

Yes X No     

Comments: None.

The continuing calibration blanks were run at 10% frequency.

Yes X No     

Comments: None.

A laboratory/preparation blank was run at the frequency of one per twenty samples, or per sample delivery group (whichever is more frequent), and for each matrix analyzed.

Yes X No     

Comments: None.

All analyzed blanks were free of contamination.

Yes      No X

Comments: The following table lists the blanks with contamination, elements present, affected samples, and data qualifiers:

TYPE OF BLANK	ELEMENTS PRESENT; CONCENTRATION	SAMPLES AFFECTED - DATA QUALIFIERS
CCB3	potassium; -917 µg/L	MHDD38 - J
	thallium; -2.9 µg/L	MHDD35 - J
CCB1	aluminum; -24 µg/L	MHDA90, MHDD37 - J*
CCB2	aluminum; 13 µg/L	MHDA90, MHDD37 - UJ
	thallium; 2.4 µg/L	MHDA90, MHDA99, MHDD37 - UJ

\* Aluminum was subsequently qualified for positive blank results. The final qualifier is "UJ".



# INORGANIC DATA QUALITY ASSURANCE REVIEW

TYPE OF BLANK	ELEMENTS PRESENT; CONCENTRATION	SAMPLES AFFECTED - DATA QUALIFIERS
MHDA96 Rinsate 09/13/95	aluminum; 42 µg/L	MHDA90 - UJ
	antimony; 4.4 µg/L	MHDA89, MHDA95, MHDA97, MHDA98 - UJ
	beryllium; 1.0 µg/L	MHDA89, MHDA91, MHDA97 - UJ
	sodium; 317 µg/L	MHDA91, MHDA95, MHDA97, MHDA98 - UJ
	zinc; 6.8 µg/L	MHDA90 - UJ

## FORM 4 - ICP INTERFERENCE CHECK SAMPLE

The ICP interference check sample (ICS) was run twice per eight hour shift and/or at the beginning and end of each sample set analysis sequence (whichever is more frequent).

Yes X No   

Comments: None.

Percent recovery of the analytes in solution ICSAB were within the range of 80-120%.

Yes X No   

Comments: None.

The ICSA and ICSAB contained no false positive or false negative results greater than the IDL.

Yes    No X

Comments: The following results greater than the IDL were reported for the interference check samples. Two samples had interferents at comparable or higher concentrations but had analyte concentrations much greater than concentrations found in the interference check samples. No sample results were qualified.

## ICSA1

ELEMENT	TRUE VALUE	IDL (µg/L)	ICSAI (µg/L)	ICSAF (µg/L)
antimony	0	3.0	4	5
barium	0	1.0	2	2
manganese	0	1.0	-3	-3
nickel	0	1.0	2	2

# **INORGANIC DATA QUALITY ASSURANCE REVIEW**

ELEMENT	TRUE VALUE	IDL ( $\mu\text{g/L}$ )	ICSAI ( $\mu\text{g/L}$ )	ICSAF ( $\mu\text{g/L}$ )
potassium	0	834	2555	2537
selenium	0	2.0	<IDL	-4
sodium	0	142	<IDL	-188
thallium	0	2.0	-2	-5
zinc	0	1.0	4	4

## **ICSA2**

ELEMENT	TRUE VALUE	IDL ( $\mu\text{g/L}$ )	ICSAI ( $\mu\text{g/L}$ )	ICSAF ( $\mu\text{g/L}$ )
antimony	0	3.0	3	6
arsenic	0	2.0	4	4
cadmium	0	1.0	-2	-2
manganese	0	1.0	-3	-3
nickel	0	1.0	2	2
potassium	0	834	3190	2618
selenium	0	2.0	-3	-4
sodium	0	142	-166	-175
thallium	0	2.0	5	<IDL
zinc	0	1.0	5	5

## **ICSA3**

ELEMENT	TRUE VALUE	IDL ( $\mu\text{g/L}$ )	ICSAI ( $\mu\text{g/L}$ )	ICSAF ( $\mu\text{g/L}$ )
copper	0	4.0	28	48
manganese	0	2.0	-4	-5
zinc	0	5.0	-7	-5

# INORGANIC DATA QUALITY ASSURANCE REVIEW

## ICSA4

ELEMENT	TRUE VALUE	IDL ( $\mu\text{g/L}$ )	ICSAI ( $\mu\text{g/L}$ )	ICSAP ( $\mu\text{g/L}$ )
arsenic	0	2.0	6	4
barium	0	1.0	-3	-3
cadmium	0	1.0	-2	-2
lead	0	1.0	-2	-2
manganese	0	2.0	-3	-3
nickel	0	1.0	3	2
potassium	0	834	3203	2039

## ICSA5

ELEMENT	TRUE VALUE	IDL ( $\mu\text{g/L}$ )	ICSAI ( $\mu\text{g/L}$ )	ICSAP ( $\mu\text{g/L}$ )
lead	0	1	-3	21*

\* Absolute value is greater than the CRDL.

## ICSAB1

ELEMENT	TRUE VALUE	IDL ( $\mu\text{g/L}$ )	ICSABI ( $\mu\text{g/L}$ )	ICSABF ( $\mu\text{g/L}$ )
antimony	0	3.0	4.4	4.6
arsenic	0	2.0	2.9	3.8
potassium	0	834	3292.6	3418.5
selenium	0	2.0	-4.1	-3.4
sodium	0	142	-234.6	<IDL
thallium	0	2.0	-3.5	-5.3

## ICSAB2

ELEMENT	TRUE VALUE	IDL ( $\mu\text{g/L}$ )	ICSABI ( $\mu\text{g/L}$ )	ICSABF ( $\mu\text{g/L}$ )
antimony	0	3.0	3	7.1
arsenic	0	2.0	3.8	3.2
potassium	0	834	3281.7	3226.4
selenium	0	2.0	-3.4	-4.2
sodium	0	142	-179.7	-175

# INORGANIC DATA QUALITY ASSURANCE REVIEW

ICSAB4

ELEMENT	TRUE VALUE	IDL ( $\mu\text{g/L}$ )	ICSABI ( $\mu\text{g/L}$ )	ICSABF ( $\mu\text{g/L}$ )
antimony	0	3.0	4.9	<IDL
arsenic	0	2.0	2.7	3.7
potassium	0	834	1802.0	2348.0
sodium	0	142	-225.9	-196.9

## FORM 5A - MATRIX SPIKE SAMPLE ANALYSIS

A matrix spike sample was analyzed with every twenty or fewer samples of a similar matrix, or one per sample delivery group (whichever is more frequent).

Yes X No     

Comments: None.

Spike recoveries were within the range of 75 - 125% (an exception is granted where the sample concentration is 4 times the spike concentration).

Yes      No X

Comments: The following table lists the spike recoveries outside control limits, samples affected, and data qualifiers.

ELEMENT	SPIKE RECOVERY	SAMPLES AFFECTED - QUALIFIERS
copper	126.3%	MHDA89, MHDA91, MHDA95, MHDA97, MHDA98, MHDB00, MHDD34, MHDD35, MHDD38 - J
lead	145.5%	
manganese	219.3%	
zinc	184.7%	

## FORM 5B - POST DIGEST SPIKE RECOVERY

A post-digest spike was performed for those elements that did not meet the specified criteria (exception: Ag, Hg).

Yes X No      N/A     

Comments: None.

**INORGANIC DATA QUALITY ASSURANCE REVIEW****FORM 6 - DUPLICATE SAMPLE ANALYSIS**

Duplicate sample analysis was performed with every twenty or fewer samples of a similar matrix, or one per sample delivery group (whichever is more frequent).

Yes   X   No       

Comments: None.

The RPDs were calculated correctly.

Yes   X   No       

Comments: None.

For sample concentrations >5 times the CRDL, RPDs were within  $\pm 20\%$  (limits of  $\pm 35\%$  apply for soil/sediments/tailings samples).

Yes        No   X   N/A       

Comments: Several elements were outside control limits for relative percent difference in the duplicate sample analysis of soil sample MHDB00. The following table lists duplicate results outside control limits, samples affected, and data qualifiers.

ELEMENT	RPD	SAMPLES AFFECTED - QUALIFIERS
aluminum	48.6%	MHDA89, MHDA91, MHDA95, MHDA97, MHDA98, MHDB00, MHDD34, MHDD35, MHDD38 - J
iron	61.2%	
lead*	46.2%	
zinc*	55.3%	

\* No additional qualifiers were added to elements previously qualified for matrix spike results.

For sample concentrations <5 times the CRDL, duplicate analysis results were within the control window of  $\pm$  CRDL (2X CRDL for soils).

Yes        No   X  

Comments: One element was outside the control window of two times the CRDL in the duplicate sample analysis of soil sample MHDB00. The following table lists duplicate results outside control limits, samples affected, and data qualifiers.

**INORGANIC DATA QUALITY ASSURANCE REVIEW**

ELEMENT	2x CRDL LIMITS mg/Kg	REPORTED DIFFERENCE (mg/Kg)	SAMPLES AFFECTED - QUALIFIERS
calcium	2203	8570	MHDA89, MHDA91, MHDA95, MHDA97, MHDA98, MHDB00, MHDD34, MHDD35, MHDD38 - J

**GFAA QC**

GFAA analyses was not performed for this SDG.

**FORM 7 - LABORATORY CONTROL SAMPLE**

The laboratory control sample (LCS) was prepared and analyzed with every twenty or fewer samples of a similar matrix, or one per sample delivery group (whichever is more frequent). An aqueous LCS is not required for mercury.

Yes X      No     

Comments: None.

All results were within the control limits.

Yes X      No     

Comments: None.

**FORM 8 - STANDARD ADDITION RESULTS**

Results from graphite furnace standard additions were correctly entered on Form I and Form VIII.

Yes           No           N/A X

Comments: None.

**FORM 9 - ICP QC**

A serial dilution was performed for ICP analysis with every twenty or fewer samples of a similar matrix, or one per sample delivery group, whichever is more frequent.

Yes X      No     

Comments: None.

# **INORGANIC DATA QUALITY ASSURANCE REVIEW**

The serial dilution was without interference problems as defined by the functional guidelines.

Yes   X        No     

Comments: None.

## **FORM 10 - QUARTERLY INSTRUMENT DETECTION LIMITS (IDL)**

IDL's were provided for all elements on the target analyte list.

Yes   X        No     

Comments: None.

Reported IDL's met contract requirements.

Yes   X        No     

Comments: None.

## **CYANIDE INSTRUMENT DETECTION LIMITS (IDL)**

An IDL for cyanide was provided in the raw data.

Yes   X        No           N/A     

Comments: None.

The reported cyanide IDL met contract requirements.

Yes   X        No           N/A     

Comments: None.

## **FORM 11 - INTERELEMENT CORRECTION FACTORS FOR ICP**

Interelement corrections for ICP were reported and met contract requirements.

Yes   X        No     

Comments: None.

## **FORM 12 - ICP LINEAR RANGES**

ICP linear ranges were reported and met contract requirements.

Yes   X        No     

Comments: None.

**INORGANIC DATA QUALITY ASSURANCE REVIEW**

**FORM 13 - PREPARATION LOG**

Information on the preparation of samples for analysis was reported on Form XIII.

Yes  X  No    

Comment: None.

**FORM 14 - ANALYSIS RUN LOG**

A Form XIV with the required information was filled out for each analysis run in the data package.

Yes  X  No    

Comments: None.

**Additional Comments or Problems/Resolutions (not addressed above).**

Water samples MHDA99 and MHDD36 were duplicate samples analyzed for total and dissolved metals respectively. The data for the two types of analyses were reviewed and results were comparable.



## **INORGANIC DATA QUALITY ASSURANCE REVIEW**

### **REGION VIII**

#### **DATA QUALIFIER DEFINITIONS**

For the purpose of Data Validation, the following code letters and associated definitions are provided for use by the data validator to summarize the data quality. Use of additional qualifiers should be carefully considered. Definitions for all qualifiers used should be provided with each report.

#### **GENERAL QUALIFIERS for use with INORGANIC DATA**

- R**     -   Reported value is "rejected". Resampling or reanalysis may be necessary to verify the presence or absence of the compound.
- J**     -   The associated numerical value is an estimated quantity because the Quality Control criteria were not met.
- UJ**    -   The reported amount is estimated because Quality Control criteria were not met. Element was not detected.

**TARGET SHEET**  
EPA REGION VIII  
**SUPERFUND DOCUMENT MANAGEMENT SYSTEM**

DOCUMENT NUMBER: 374980

SITE NAME: RICO ARGENTINE/RICO POND

DOCUMENT DATE: 11/06/1995

**DOCUMENT NOT SCANNED**

Due to one of the following reasons:

- ☐ PHOTOGRAPHS
- ☐ 3-DIMENSIONAL
- ☐ OVERSIZED
- ☐ AUDIO/VISUAL
- ☐ PERMANENTLY BOUND DOCUMENTS
- ☐ POOR LEGIBILITY
- ☐ OTHER
- ☐ NOT AVAILABLE
- ☒ TYPES OF DOCUMENTS NOT TO BE SCANNED  
(Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:

INORGANIC ANALYSES DATA SHEETS

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